SECTION 1 COURSE OBJECTIVES AND INTRODUCTION TO CRYOGENS

COURSE OBJECTIVES

This course will provide basic information concerning cryogens and their use at the NHMFL. As a result of this training, the attendee will be able to:

- Define important terms including cryogen, critical point, critical temperature, vacuum jacket, pressure relief device, dewar, thermal stress, air condensation, and cold embrittlement.
- Identify liquid nitrogen and helium as the primary cryogens used at the NHMFL.
- Explain the pressure hazards associated with contained cryogenic fluids.
- Explain techniques used to control hazards associated with cryogenic fluids.
- Describe a cryogen containment system.
- Identify a "Dewar" and how to properly use it.
- Identify the major physical/health hazards associated with cryogens which include air condensation, cold embrittlement, asphyxiation, and skin/eye contact.
- Identify first aid procedures for treating cryogenic burns.

N O T E S:

- Identify cryogen guidelines, safety procedures (SPs), and operating procedures (OPs) established at the NHMFL.
- Identify danger signs that indicate hazardous conditions within or around a cryogen containment system.
- Identify appropriate procedures for reporting cryogen accidents and incidents.

WHAT IS A CRYOGEN?

A cryogen is an extremely cold element or compound. Cryogens are liquefied gases which have boiling points below 222°K or -238°F (source "Encyclopedia of Occupational Health and Safety"). That means at room temperature, and under normal conditions, cryogens will rapidly begin to boil and convert from a liquid to a gas.

WHAT CRYOGENS ARE USED AT THE NHMFL

The NHMFL routinely handles two cryogenic liquids, helium and nitrogen. Liquefied nitrogen has a boiling point, at atmospheric pressure, of 77°K (-320°F). Liquefied helium has a boiling point of, at atmospheric pressure, of 4.3°K (-452°F). At one atmosphere pressure the liquid temperature is the same as the boiling point.

N O T E S :

CRYO is a Greek word for "icy cold". GEN is a Greek root meaning "to make". So CRYOGEN refers to a substance used to make other things cold.

Extremely low temperatures are reported using units called Kelvin (K). For reference, O°K = -273°C = -460°F (also called absolute zero).

Boiling Points for NHMFL Cryogens

	°F	*°C*	*°K*
Nitrogen	*-320*	*-195*	*77*
Helium	*-452*	*-269*	*4.3*

SECTION 1 COURSE OBJECTIVES AND INTRODUCTION TO CRYOGENS

HOW ARE CRYOGENS USED AT NHMFL?

Cryogens are routinely used for the cooling of magnets and experiments. The magnets are pre-cooled using nitrogen, and then cooled with helium to their final temperature. Nitrogen is used for pre-cooling because it is less expensive than helium and has a higher heat capacity which means more danger during physical contact.

Cryogens are also used in the physical testing of various materials. Experiments are conducted to evaluate how certain materials respond to extremely cold temperatures. For example, some materials show super conducting capabilities when cooled by cryogens.

THE IMPORTANCE OF CRYOGEN SAFETY

Cryogenic materials are used at this facility. If not handled properly, cryogenic fluids can be hazardous to personnel. By participating in this cryogen safety training program, you will become aware of the conditions that increase the risk of accidents and injuries that can happen when working with cryogenic fluids. What you learn will make your work place safe for you and your co-workers.

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SECTION 2 PHYSICAL PROPERTIES AND CONTROL MEASURES

Cryogens have physical properties which are not typical of the materials that are found at room temperature and standard atmospheric pressure.

CRITICAL POINT AND CRITICAL TEMPERATURE

The critical point, not to be mistaken with boiling point, is the highest temperature at which a material changes from a gas to a liquid regardless of pressure. The temperature at which this takes place is called the critical temperature.

The cryogenic fluids used at the NHMFL, namely nitrogen and helium, have very low critical temperatures, 126.3° K(-232°F) for nitrogen and 5.2° K (-450°F) for helium.

CRYOGENIC FLUID VAPORIZATION

Due to the extremely low critical temperatures, when cryogenic fluids are heated (i.e., exposed to room temperature) they turn into a gas very rapidly. If cryogenic fluids are confined inside a container the pressure from the vaporization of the liquid can be great.

CONTROLLING PRESSURE RELEASES

To eliminate high pressure releases of cryogenic vapors, containment systems with special pressure-relief devices are used. They typically consist of pressure relief valves and/or breakable “Burst Disks” to allow over pressures to release safely. Also some containment systems have values which are held in by atmospheric pressure and will eject forcefully during an accident.

N O T E S :

Remember, cryogens are gases under standard temperature and pressure.

When exposed to room temperature, liquid nitrogen can generate a pressure of 45,000 pounds per square inch (PSI). Normal atmospheric pressure is 14.7 PSI.

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Note: Liquid helium has a 700:1 expansion ratio of gas to liquid.

CONTAINMENT SYSTEM

The containment systems used for cryogenic liquids includes an insulated container where the cryogenic liquid is stored, delivery lines, and a vacuum jacket.

The typical container used to store and handle cryogenic fluids at the NHMFL is the dewar. The dewar is designed with a vacuum jacket for insulation and pressure relief valves to protect against over-pressurization (see Figure 2-1).

VACUUM JACKETS

The vacuum jacket acts as an insulating layer for the cryogenic fluid and sometimes includes a system of coiled pipes between the inner and outer walls. The vapors from the cryogenic fluid circulate through the piping system and cool the inner and outer walls of the containment.

The vacuum jacket protects workers from the extreme cold of the cryogenic liquid and protects the cryogenic fluid from the ambient temperature of the surrounding environment.

PRESSURE RELIEF DEVICES

The pressure relief devices found on the dewar usually consists of spring-loaded valves and burst disks. These types of pressure relief devices should be used on any component of the containment system where cryogenic liquid is enclosed, including all delivery lines and cut-off valves.

Liquid helium dewars are designed with two over-pressure relief valves and an over-pressure rupture disk. The over-pressure reliefs are initiated at 0.5 PSI 10 PSI at room temperature, respectively. The 0.5 PSI relief value can be used to perform liquid transfers. If the vessels pressure increases to 38 PSI, the rupture disk will relieve vessel pressure to the atmosphere. Liquid nitrogen dewars have one pressure relief valve set at 22 PSI and a rupture disk set at 189 PSI.

PROPER USE OF DEWARS

All cryogen containers (dewars) should be operated in accordance with the manufacturer's instructions. Proper personal protective equipment must be worn whenever handling cryogenic liquids.

Dewars are designed to protect workers from contacting cryogenic fluids and maintain the cryogen in its liquefied state. However, proper Dewar handling practices must be used to ensure worker safety. Safe Dewar handling practices include:

- Never cross contaminate in service Dewars with other cryogenic liquids.
- Ensure dewars are properly labeled with the identity of the housed cryogen.
- Keep the dewar upright. Do not bump or drop the dewar from an elevation. This could ruin the insulating properties of the dewar. Dewars that fall onto their side could rupture if the inner vessel cracks and cryogenic material flows into the vacuum space between the inner and outer vessels. The cryogen will contact the warm metal and boil

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Note: The NHMFL uses heaters to pressurize dewars to 4 PSI to facilitate material transfers.

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rapidly, greatly increasing the pressure in the dewar.

- When using a hand truck or fork truck for dewar transport, the dewar must be strapped onto a dewar transport pallet. Never use chains to secure the dewar to the transport pallet.
- Never slide or roll a dewar.
- When using a crane to lift Dewars, use approved lifting devices specifically designed for dewars. Never lift dewars by their handles or by means of slings wrapped around the shell of the dewar.
- Use cryogen rated personal protective equipment when filling, venting, and transferring dewars and cryogenic fluids.
- Ensure dewars are positioned so that the pressure relief valves and rupture disks vent paths are directed away from personnel, critical equipment, or designated work areas.
- The fill and vent ports of the dewar should be kept closed at all times to minimize the formation of ice which may plug the pressure relief devices. The pressure relief devices should be periodically inspected for ice.
- If dewar vent posts are shut, periodically monitor dewar pressure. If indicated pressure exceeds 15 PSI, relieve pressure by cycling vent valve.

- When filling dewars or transferring cryogenic liquid, use a phase separator or special filling funnel. The top of the funnel should be partly covered to reduce splashing. If the liquid cannot be poured, use a cryogenic liquid withdrawal device for the transfer. Be sure to follow all instructions provided with the device.
- Additionally, all cryogenic equipment used at the NHMFL shall be maintained in accordance with manufacturer's recommendations. Any equipment not meeting manufacturer's operating specifications shall be removed from service.

THERMAL STRESS

The parts of a cryogen containment system sometimes experience great differences in temperature. These stresses, known as *thermal stresses*, can cause many problems for poorly designed containment systems.

The design of a cryogenic containment system must take into consideration the normal thermal contraction of the containment system when exposed to the temperature difference between the inside (cryogenic liquid) and the outside of the containment system (room temperature).

As a cryogenic liquid proceeds down the length of the pipe, the piping material experiences changes and stress resulting from the extreme temperature differences between the inside and

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Example: The helium gas recovery system plumbing is made of plastic pipes that are not designed to handle thermal stress.

outside of the pipe. The degree of stress and potential for failure depends upon the properties of the pipe material and the flow rate of the cryogenic liquid.

CONTROLLING THERMAL STRESS

The following steps should help you minimize the risk of failure due to thermal stress:

- When starting up the system, allow for gradual cooling of the system.
- Use cryogen approved materials for the containment system.
- Select materials and equipment that can accommodate the causes and effects of thermal stress.

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HAZARDS CAUSED BY AIR CONDENSATION

There is always a chance that the air surrounding a cryogen containment system can condense especially when transferring liquid nitrogen through uninsulated metal pipes or relieving pressure in liquid helium dewars. Air condensation can cause hazards to workers and equipment. These include the following:

- It can create a liquid condensate that falls on materials, particularly organic materials, susceptible to cold embrittlement.
- It can increase the oxygen concentration around a containment system, which can increase the flammability of materials near the system. For example, nitrogen, which has a lower boiling point than oxygen, will evaporate first, leaving an oxygen enriched condensate on the surface.
- Clothing saturated with oxygen from air condensed by cryogenic fluids readily ignites and will burn vigorously. Personnel in this situation should immediately leave the area and avoid all ignition sources.

Based on air condensation effects, equipment containing cryogenic fluids must be kept clear of combustible materials in order to minimize the fire hazard potential.

CONTROLLING AIR CONDENSATION

The frost that accumulates on containment system components is very important in

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Example: The millikelvin pits contain magnets which are cooled with liquid nitrogen then liquid helium. The liquid nitrogen and helium could displace the oxygen inside the pit enough to cause an asphyxiation hazard to anyone who enters the pit.

controlling air condensation. It insulates the containment system from the surrounding air, reducing the possibility that the air will condense.

Another way of reducing air condensation is by applying an insulating material on system components. This not only reduces air condensation but also protects workers by preventing contact with cold cryogenic containment system surfaces.

COLD EMBRITTLEMENT

Many materials become very fragile at very low temperatures. At cryogenic temperatures (below 222°K), materials such as rubber, plastic and carbon steel can become so brittle that very little stress can break the material.

To avoid problems with cold embrittlement, materials like stainless steel, copper, brass and most alloys of aluminum should be used in the cryogen containment systems.

ASPHYXIATION HAZARD

Cryogens will rapidly boil and convert from a liquid to a gas at room temperature. As the gas warms to the temperature of the surrounding air, it expands. In confined or poorly ventilated areas, the expanding gas will displace oxygen and can cause rapid asphyxiation or death. Therefore, you should use caution when using liquid nitrogen and helium indoors. These gases are colorless, odorless, and tasteless.

ASPHYXIATION WARNING SIGNS

OSHA specifies that workers cannot be inside a work space that contains less than 19.5% oxygen without supplied air respiratory protection. Below this level, workers start to experience early warning signs of oxygen deficiency. Specifically between 15-19% oxygen workers may feel:

- A loss of coordination and energy.
- An increase in pulse rate and breathing.
- A sense of euphoria and clumsiness.

At oxygen levels between 12-14% the worker's:

- Breathing becomes much deeper and faster.
- Judgment becomes impaired.
- Physical coordination is deteriorated.
- Lips turn blue.

Usually at levels below 12% the worker will become unconscious and eventually die.

PREVENTING ASPHYXIATION HAZARDS

When using cryogens indoors, make sure the room is well ventilated and you have the cryogen stored in proper containment systems.

If cryogens are used in enclosed or poorly ventilated work areas, confined space entry procedures may be needed. If this is the case, the following work practices should be followed:

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<u>*Examples of areas where oxygen deficient atmospheres could exist*</u>:

Helium Recovery Room
Hybrid Platform
Millikelvin Labs Pits
CIZO Lab Pits

- Never enter a confined space without an attendant or safety watch present.
- Determine the oxygen level inside the enclosed space before entry to make sure oxygen levels are within the safe oxygen limits of 19.5-23.5%. Air supplied respiratory protection must be worn in any space with less than 19.5% oxygen. Self contained breathing apparatus (SCBA) are located next to the Control Room for use by trained personnel.
- Areas suspected of being oxygen deficient due to the release of large quantities of cryogenic vapors shall be evacuated immediately.
- If necessary, ventilate the work space with clean (breathable) air to establish oxygen concentrations within the safe limits.
- Evaluate any other hazards inside the confined space such as flammable gasses, toxic gases, electrical equipment, heat, cold, etc.
- Fill out and follow all control measures defined in the Confined Space Entry Permit, if they apply to your situation. All personnel entering the confined space should be properly trained according to the OSHA Confined Space Standard.
- When working with cryogens inside an enclosed area, you should continuously monitor the air for oxygen concentrations. This will provide early

warning to workers if oxygen levels fall below safe levels.

SKIN AND EYE HAZARDS

Never make direct contact with cryogenic liquids, uninsulated cryogenic pipes, or uninsulated cryogenic equipment because contact can cause freeze burns and tissue damage. In addition, a jet of cryogen vapors can freeze the skin or eyes faster than liquid contact, even faster than metal contact.

The eyes contain fluids and are especially sensitive to cryogen exposure. These fluids will freeze upon contact with a cryogen causing permanent eye damage. Exposure which does not damage the skin may cause permanent eye damage.

PERSONAL PROTECTION

The precautions listed below should be followed to protect the eyes and skin from the hazards associated with cryogens.

- Insulate all containment system pipes.
- Use care when filling portable Dewars.
- Always wear protective gloves over jewelry because if exposed to cryogenic fluids, the ring can freeze to the finger.
- Protect your eyes by wearing safety goggles or a face shield whenever working with cryogen fluids.
- Wear a cryogen apron when working with cryogen liquids.

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- Always wear a face shield when working around pressurized cryogenic systems, connecting or disconnecting cryogenic equipment lines, or when venting containment systems.
- Wear insulated gloves whenever working around uninsulated pipes or handling Dewars.
- Try to cover all exposed skin by wearing long sleeve shirts, cuffless pants, safety boots, and gloves. Gloves should be loose-fitting so that they can be quickly removed if cryogenic fluids are spilled on them.

FIRST AID PROCEDURES FOR CONTACT

Any time cryogens come in contact with your skin, you should obtain medical assistance as soon as possible.

Immediately after exposure, the frozen skin appears waxy and yellow and usually is not painful to the worker. As the skin thaws, it painfully swells and blisters. When this occurs, immediate emergency treatment is required. While waiting for medical assistance, follow these first aid procedures:

- Remove the victim from the cryogen hazard.
- Remove any clothing that may interfere with the circulation of blood to the frozen tissues. The clothing must be removed in a slow, careful manner to prevent salvageable skin from being pulled off.

- Do not rub the affect areas of skin. Rubbing may further damage the tissue.

- Immerse the effected area in a warm water bath (approx. 105°F). Do not apply dry heat, such as electric heaters, because it may superimpose a thermal burn, further damaging injured tissue.

- If the worker has experienced a massive exposure such that overall body temperature is reduced, the worker should be wrapped in blankets until paramedics arrive. In cases of extreme exposure, the worker should be totally immersed in warm water. Treatment for shock may be necessary.

- The rewarming, or thawing, of affected area(s) should be done gradually. It may take up to 60 minutes to thaw the affected area(s) and bring back the natural colors of the skin.

- If the frozen tissue thaws before medical help arrives, cover the area with dry sterile dressings and large bulky protective clothing. Do not apply ointments.

- Do not allow the exposed worker to drink alcohol or smoke. Alcohol and nicotine decrease blood flow to the frozen tissues. You may give the effected worker warm drinks and food.

- Try to make the worker feel as comfortable as possible.

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Minor burns must be reported to the NHMFL Safety Department and your supervisor. Obtain medical attention if appropriate.

SECTION 4 CRYOGEN SAFETY PROCEDURES

THE NHMFL CRYOGEN POLICIES

Prior to using cryogenic liquids, users shall familiarize themselves with procedures provided by the NHMFL on the safe use and handling of cryogenic fluids. These procedures and guidelines include:

- CRYOGEN FLUID SAFETY GUIDELINES for laboratory users and employees (Jan. 1994).
- CRYOGENIC SAFETY FOR LABORATORY USERS, SP-4.
- DEWAR FILLING OPERATIONS, OP-4.
- DEWAR DISK RUPTURE, SP-6.
- LIQUID NITROGEN STORAGE TANK RELIEF DISK ACTUATION, SP-5.

These guidelines and procedures can be found in Attachment A of this manual.

NHMFL personnel shall notify the Control Room immediately of any accident or abnormal situation involving cryogens. Control Room personnel will provide immediate assistance to control the situation and will contact the NHMFL Safety Department for assistance.

The following subsections provide a summary of each of the NHMFL's Safety Procedures (SP) and Operating Procedures (OP) addressing the use and safe handling of cryogens.

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CRYOGEN FLUID SAFETY GUIDELINES

This guideline summarizes cryogenic fluid safety precautions which must be followed by all personnel working with these fluids and systems. Specifically, the guideline describes:

- Safety precautions which must be observed when working with liquid helium and liquid nitrogen.
- Personal protective equipment and procedures that can be used to protect against contact with cryogenic fluids.
- How to operate cryogenic containers (Dewars) in a safe manner.
- First aid procedures for cold burns from cryogens.

CRYOGENIC SAFETY FOR LABORATORY USERS, SP-4

This SP presents the policies and procedures to be followed by all users of cryogenic liquids during experimentation. This SP includes responsibilities, provides safety guidance, and defines actions to be taken in the event of an accident or release involving cryogenic liquids.

DEWAR FILLING OPERATIONS, OP-4

This OP defines operational guidelines and safety concerns to be observed by personnel when filling dewars with liquid helium and nitrogen. This OP is to be used by all personnel who are qualified to operate dewar fill stations.

The liquid helium dewar fill station is a part of the Helium Recover, Purification, and Liquefaction System. This fill station is located at the rear of the OP/MD building, near the Hybrid Magnet Cell.

The liquid nitrogen dewar fill station is also located at the rear of the OP/MD building, near the Hybrid Magnet Cell.

DEWAR DISK RUPTURE, SP-6

This SP defines procedures to be used in the event of a dewar pressure relief disk rupture. This SP identifies specific reporting procedures and actions required by NHMFL personnel and users following over-pressurization and subsequent rupture of a dewar pressure relief disk.

LIQUID NITROGEN STORAGE TANK RUPTURE DISK ACTUATION, SP-5

This SP defines procedures to be used in the event the Liquid Nitrogen Storage Tank pressure relief rupture disk actuates. This SP identifies specific reporting procedures and actions required by NHMFL personnel and users following over-pressurization and subsequent rupturing of the Liquid Nitrogen Storage Tank rupture disk.

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SECTION 5 EMERGENCY PROCEDURES

The release of cryogens from their containers may require emergency procedures. Recognizing signs of problems with the containment system can prevent emergencies or let people solve the problems quickly.

CONDITIONS THAT INDICATE PROBLEMS

Workers who handle cryogenic fluids must be aware of conditions, or danger signs, that may indicate the buildup of excess pressure in cryogenic systems. These conditions may include:

- Elevated pressures indicated on gauges.
- Unexpected frost formation on containment systems.
- Poor or abnormal venting in the containment system.
- Warning alarms indicating low levels of oxygen in the work area.
- Unusual noise or absence of usual venting noises.

DEWAR DISK RUPTURES OR VENTING OF LARGE AMOUNTS OF GAS OR LIQUID

Signs that a large amount of cryogen is being vented include increased background noise levels and condensation of air around the escaping gas into a white, fog-like plume.

N O T E S :

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When a large volume of excess internal liquid or gas is released to the atmosphere, the immediate vicinity of the dewar will have a high concentration of low temperature vapor. As stated in Section 2, released cryogenic vapors pose a great potential for asphyxiation due to oxygen deprivation in areas of poor air circulation. However, the most likely immediate personnel hazard associated with dewar disk rupture is frostbite burns resulting from contact with low temperature gas or liquid.

DISK RUPTURE SAFETY PROCEDURES

In the event that a dewar releases cryogenic liquids to the atmosphere via a ruptured disk:

- All personnel in the immediate vicinity of the release should be informed of the dewar disk rupture.
- All personnel in the immediate vicinity are required to evacuate to a safe distance.
- The disk rupture must be reported to the magnet cell Control Room and NHMFL Safety Department immediately by the person filling the dewar or anyone witnessing the disk rupture.

Examples of Minor Leaks

Spilling a small dewar or flask.
Leak in the fill line.
Leak in the transfer line.

MINOR LEAK RESPONSE

In the event of a minor leak from a dewar, the following steps must be taken:

- If filling a dewar, shut down the containment system.

- Disconnect the dewar from the system without contacting the cryogenic fluids. If the dewar cannot be disconnected without contacting the liquid, evacuate the affected area.
- Once disconnected, remove the dewar to the nearest door and allow the vapor to be released to the atmosphere.

MAJOR LEAK RESPONSE

In the event of a major leak from a dewar, the following steps must be taken:

- Immediately evacuate the area containing the dewar establishing a safe distance or buffer zone.
- If you are in an energized magnet cell, push the EPS emergency shutdown switch and command magnet to ramp to zero quickly before leaving cell.
- Report the dewar leak and any injuries or illness to the magnet cell Control Room Operator immediately. There are times when the Control Room Operator may not be immediately accessible. In this case, contact the NHMFL Safety Department. The following Safety Department staff can be contacted at:

  Safety Coordinator
  (904) 644-0233 Office
  (904) 657-8278 Pager

N O T E S :

Examples of Major Leaks

Magnet quench.
Liquid nitrogen and helium dewar ruptures.
Nitrogen storage tank failure.
Helium recovery room bags over-inflating.

Magnet Quench

Magnet Quench occurs when a superconducting magnet becomes resistive and can't recover back to superconductivity. This can result from loss of cryogen coolant, introduction of a magnetic field, or exceeding field limitations. Current to the magnet is transferred to a resistor or to the power supply. The liquid coolant boils to a gas and is released via pressure relief valves or recovery lines.

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Hazardous Materials Manager
(904) 644-6955 Office
(904) 657-8279 Pager

- Try to determine whether there is adequate ventilation in the affected area. If it cannot be determined, inform the Control Room Operator and he will ensure that adequate ventilation is provided.

- Never re-enter the area until it has been determined "Safe" by the Control Room Operator, Safety Coordinator, or Hazardous Materials Manager.

In this program, we defined the term “cryogen” and identified which cryogens are used at the NHMFL, namely liquid nitrogen and helium. This program also reviewed how cryogens are used at the NHMFL. The physical properties of cryogens and the control measures required for safe use were discussed. Health hazards and protective measures were identified. The NHMFL has several cryogen policies and procedures that were reviewed during this program. This program also reviewed applicable emergency procedures for the unplanned and uncontrolled release of cryogens from their containers.

Cryogens are critical to the operation of the NHMFL. While these materials do have inherent hazards, they can be used safely if the appropriate procedures are followed and control measures implemented.

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